TABLE 1

SELECTED LIST OF SPECIES DETECTED

Class species	Photoionization Response 9.5eV lamp	10.2 eV lamp	11.7 eV lamp
Paraffins and unsaturated hydrocarbons			
methane	NR	NR	NR
ethylene	NR	L	H
acetylene	NR	NR	H
1-butene	H	H	H
hexane	Nr	L	H
Chlorinated hydrocarbons			
methyl chloride	NR	NR	H
carbon tetrachloride	NR	NR	H
chloroform	NR	NR	H
dichloroethane	NR	NR	H
vinylidene chloride	L	H	H
trichloroethylene	H	H	H
Heterocyclics & aromatics			
phenol	H	H	H
pyridine	H	H	H
benzene	H	H	H
toluene	H	H	H
xylene	H	H	H
styrene	H	H	H
aniline	H	H	H
chlorobenzene	H	H	H
nitrobenzene	NR	L	H
Nitrogen compounds			
formamide	NR	H	H
ammonia	NR	L	H

TABLE 1 (Continued)

Class species	Photoionization Response 9.5eV lamp	10.2 eV lamp	11.7 eV lamp
Nitrogen compounds (Continued)			
hydrazine	H	H	H
methyl amine	H	H	H
acetonitrile	NR	NR	NR
acrylonitrile	NR	NR	H

NR = No response.
H = High response.
L = Low response.

B.) OPERATION

1.) Before attaching the probe, check the function switch on the control panel to make sure it is in the "OFF" position. Figure 1.

2.) Carefully match the alignment key in the probe connector to the 12 pin connector on the control panel, and then twist the probe connector until a distinct snap and lock is felt.

3.) Turn the function switch to the battery check position. The needle on the meter should read within or above the green battery arc on the scale plate.

If the needle is in the lower portion of the arc, recharge before use. If the LED comes on, recharge before use. (See Section D. Recharging the HNU.)

4.) Turn the function switch to "On." In this position, the UV light source should be on. If looking into the end of the probe reveals a purple glow, the UV light source is working.

5.) Set the span to the desired gain.

6.) Zero the instrument by turning the function switch to the stand-by position and rotate the zero potentiometer knob. Clockwise produces an up-scale deflection and counterclockwise yields a downscale deflection:

NOTE: If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted, if necessary. (Stand- by position)

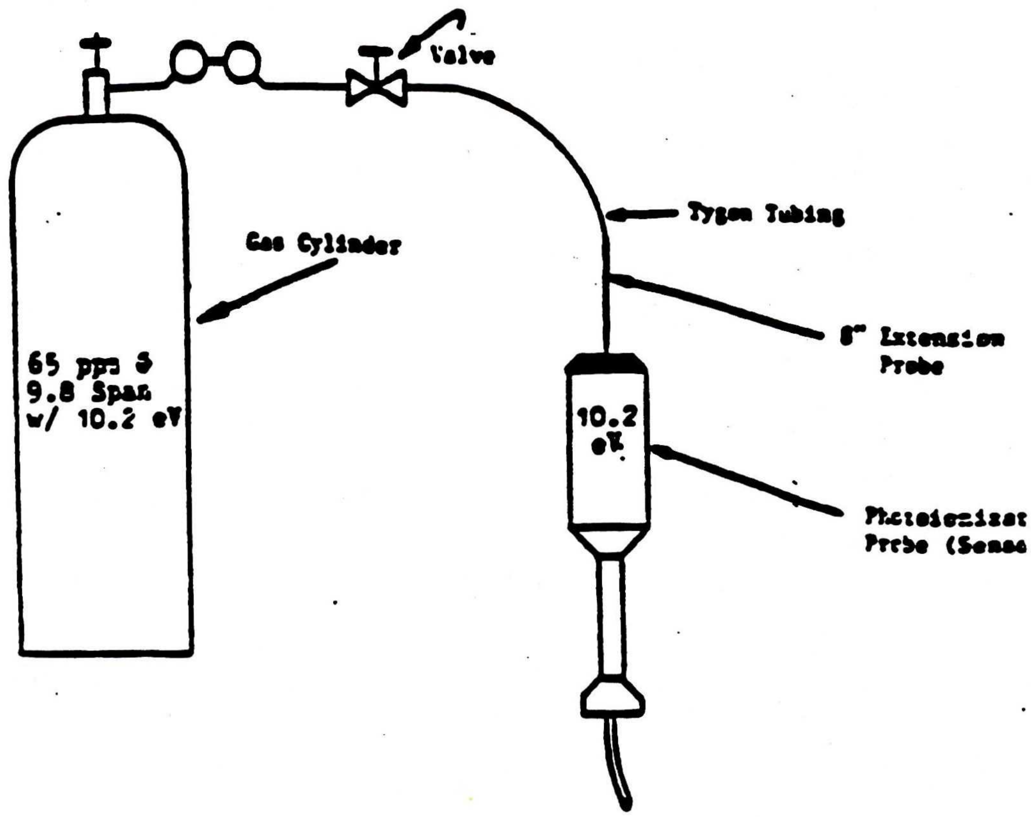

FIGURE 2
Recommended Calibration Procedure

The instrument is supplied calibrated to read directly in ppm (0-20, 0-200, 0-2000) of benzene with the span position set at 9.8. For additional sensitivity, the span poteniometer is turned counterclockwise (smaller numbers) to increase the gain. Changing the gain changes instrument sensitivity and specificity (if changed from 9.8, it will no longer be direct reading for benzene). By changing the span setting from 10.0 to 1.0, the sensitivity is increased approximately ten-fold. The 0-20, 0-200 and 0-2000 scales become 0-2, 0-20 and 0-200, respectively.

The span control can be utilized to calibrate nearly any compound, measured by photoionization, to be direct reading on the 0-20 ppm range. For example, gain settings of 4.5 or 8.9, respectively, will provide direct reading capability (0-20, 0-200 ppm) for vinyl chloride and trichloroethylene, respectively. Table 2 is a listing of approximate gain setting values for some common compounds. Note that these settings are approximate until the meter is calibrated against the specific compound.

TABLE 2

RELATIVE PHOTOIONIZATION SENSITIVITES FOR VARIOUS GASES

Grouping	Span/Gain Setting	Examples
Aromatic	9.8	Benzene, Toluene, Styrene
Aliphatic Amine	9.8	Diethylamine
Chlorinated Unsaturated	5-9	Vinyl Chloride, Vinylidene Chloride, Trichloroethylene
Carbonyl	5-7	MEK, MIBK, Acetone, Cyclohexene
Unsaturated	3-5	Acrolein, Propylene, Cyclohexene, Allyl Alcohol
Sulfide	3-5	Hydrogen Sulfide, Methyl Mercaptan
Paraffin (C_5-C_7)	1-3	Pentane, Heptane
Ammonia	0.3	--
Paraffin (C_1 - C_4)	0	Ethane, Propane, Butane

7.). The instrument is now ready for calibration or measurement by switching the function switch to the proper measurement range, i.e., 0-20, 0-200, or 0-2000.

C.) <u>CALIBRATION</u>

The recommended and most accurate procedure for calibration of the HNU instrument is utilizing a pressurized gas cylinder containing a known ppm value at a specified span setting attached to a designated probe. The following procedure refers to Figure 2.

1.) Follow steps 1-7 in OPERATION section (above).

2.) Attach the tygon tubing to the 8" extension probe of the photoionization probe.

3.) Crack the valve of the pressurized cylinder until a slight flow of gas is being released from the cylinder.

The instrument should read $\pm$10% of the gas value; if not, one of two things can be done:

a. Change span to get the gas value. NOTE: If span is changed more than $\pm$10%, proceed to b.

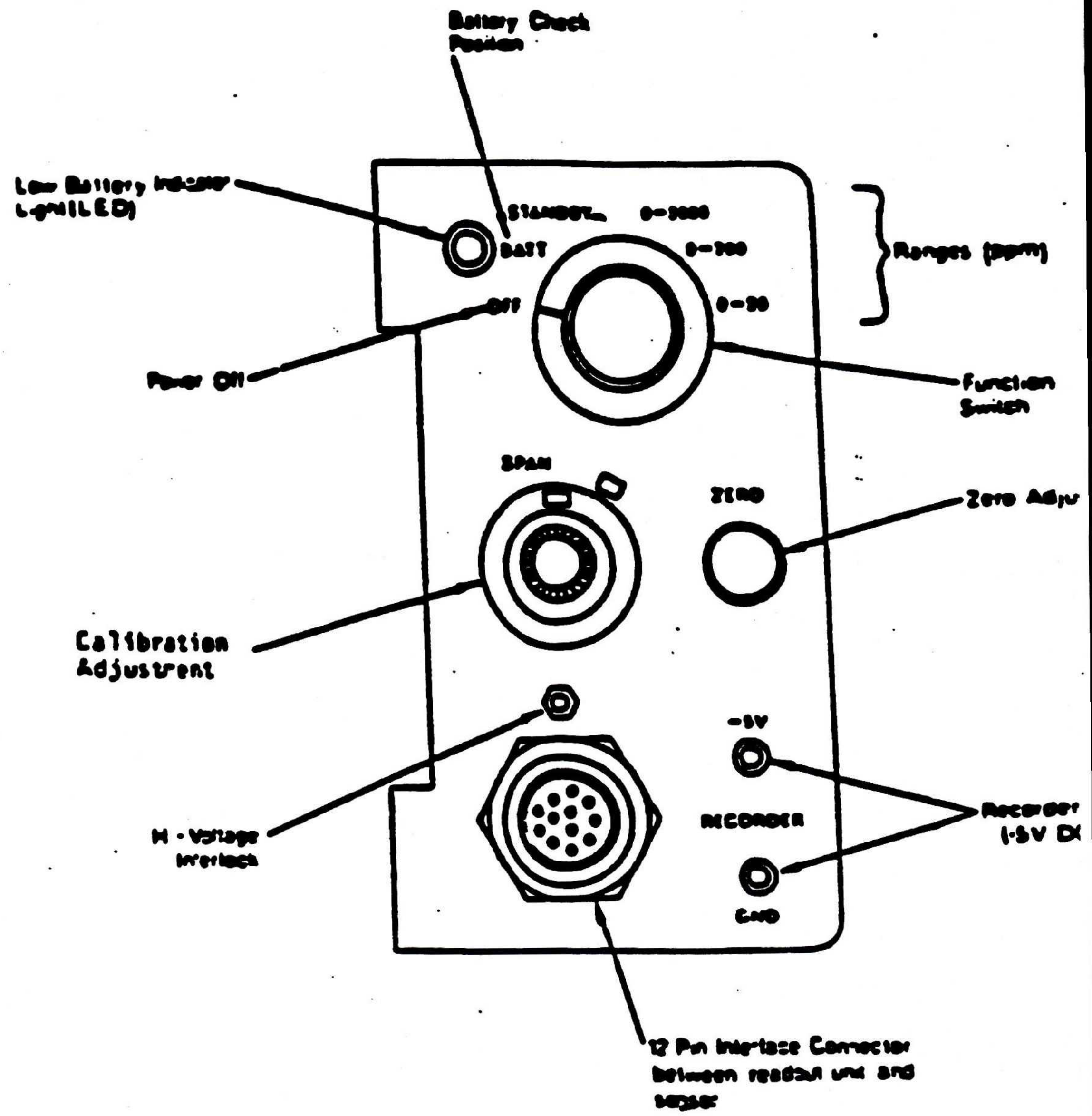

FIGURE 1
Control Panel Functions

b. Clean lamp and IP chamber. A dirty lamp will yield low readings, and a dirty chamber will yield high readings.

NOTE: If the instrument span setting is changed, the instrument should be turned back to the stand-by position and rezeroed, if necessary.

If using the 11.7 eV probe and the gas calibration cylinder showing a ppm value of 9.8 span with 10.2 eV probe, all steps above will be necessary. The final span setting using the 11.7 eV probe should be approximately the same value as indicated for the specific probe in Appendix 2.

The HNU instrument is now ready for field measurements.

D.) RECHARGING THE HNU

To ensure no damage to the HNU instrument and to extend the life of the battery, the following steps should be followed when recharging the HNU.

1.) Place the miniphone plug into the jack on the left side of the read-out unit.

2.) Plug the charger into a 120 vac outlet.

3.) Let stand overnight or for at least 14 hours.

NOTE: Overcharging is not a major problem with the HNU as it has a built-in solid-state battery protection circuit; also, when the battery voltage drops below approximately 11 volts, this circuit will automatically turn off power to the instrument. This prevents deep discharging of the battery.

It is, however, recommended that if the battery check shows ample power available, not to charge the unit.

4.) When disconnecting the charger, remove the charger from the 120 vac before removing the miniphone plug.

The instrument can be operated during the recharging cycle. This will only lengthen the time required to completely recharge the instrument battery.

E.) FALSE READINGS

Incorrect values may be detected by the HNU, outside of mechanical failures within the unit. Some of the field situations which may be encountered are as follows:

1.) High wind
2.) High humidity (>95%)
3.) Probe too far from source
4.) High electrical areas
5.) Temperatures above 105°F or below 32°F.

High wind and high humidity are two variables beyond control of the instrument operator. The probe being too far from the source is self-explanatory for correction. When working around high electrical areas, the following steps may be utilized to obtain relevant measurements.

1.) Zero the instrument in an electrically quiet area in the stand-by position.

2.) Move the instrument to the area in question. If AC pick-up is going to be a problem, the meter (in the stand-by position) will indicate the magnitude of error.

3.) Subtract this difference, Step 2, from the indicated value to obtain the actual value.

The HNU should not be used in temperatures greater than 105°F. In temperatures less than 32°F, the unit should function properly as long as the probe extention and probe inlet are wiped dry after use. The probe extension should be wiped dry because when moving the unit from a warm area to a cold area and back to a warm area again, condensation will develop inside the extension probe causing erratic values. If moisture enters into the lamp area of the probe, the following steps should be taken to free the unit of moisture or dust particles. Figure 3.

1.) Turn the function switch to the off position.

2.) Disconnect the probe from read-out unit.

3.) Remove the exhaust screw found near the base of the probe.

4.) Grasp the end cap in one hand and the probe shell in the other; gently pull to separate the end cap and lamp housing from shell.

5.) Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing.

6.) Turn the end cap over into your hand and tap on the top of it; the ion chamber should fall out into your hand.

7.) Place one hand over the top of the lamp housing and tilt slightly, the light source will slide out into your hand.

8.) Wipe dry all parts with a soft dry cloth, except for lamp and lamp window.

CAUTION: If the window in lamp of the 11.7 eV lamp is dirty and needs to be cleaned, do not clean with water or any organic solvent miscible with water such as acetone or methanol. The window should be cleaned with a soft tissue dipped in an organic (nonwater miscible) solvent or freon. The cleaning compound for the 10.2 eV lamp should not be used under any circumstances on the 11.7 eV lamp.

F.) TROUBLESHOOTING

Some of the basic problems which may occur and probable causes and solutions are as follows:

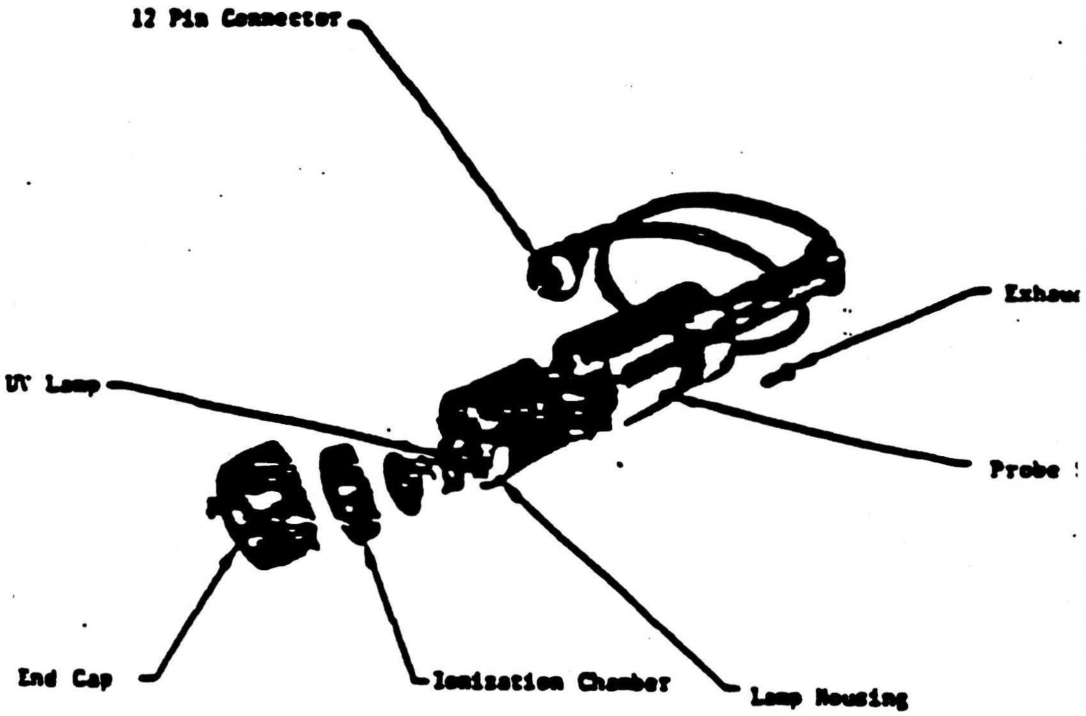

FIGURE 3
Component Parts of Probe

1. No meter response in any switch position (including BATT CHK)

   A. Broken meter movement

      (1) Tip instrument rapidly from side to side. Meter needle should move freely and return to zero.

   B. Electrical connection to meter is broken

   C. Battery is completely dead

      (1) Disconnect battery and check voltage with a volt-ohm meter.

   D. Check 2 amp fuse

   E. If none of the above solves the problem, consult the factory.

2. Meter responds in BATT CHK position, but reads zero or near zero for all others

   A. Power supply defective

      (1) Check power supply voltages. If any voltage is out of specification, consult the factory.

B. Input transistor or amplifier has failed

(1) Rotate zero control; meter should deflect up/down as control is turned.

(2) Open probe. Both transistors should be fully seated in sockets.

C. Input signal connection broken in probe or read-out

(1) Check input connector on printed circuit board. Should be firmly pressed down.

(2) Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object.

(3) Check all wires in read-out for solid connections.

3. Instrument responds correctly in BATT CHK, and STBY, but not in measure mode.

A. Check to see that light source is on.

(1) Check high voltage power supply.

(2) Open end of probe; remove lamp and check high voltage on lamp contact ring.

(3) If high voltage is present at all above points, light source has most likely failed. Consult the factory.

4. Instrument responds correctly in all positions but signal is lower than expected.

A. Check span setting for correct value.

B. Clean window of light source.

C. Check power supply 180 V output.

D. Check for proper fan operation. Check fan voltage.

E. Rotate span setting. Response should change if span pot is working properly.

5. Instrument responds in all switch positions, but is noisy (erratic meter movement).

   A. Open circuit in feedback circuit. Consult the factory.

   B. Open circuit in cable shield or probe shield. Consult the factory.

6. Instrument response is slow and/or irreproducible.

   A. Fan operating improperly. Check fan voltage.

   B. Check calibration and operation. See Sections B and C.

7. Low battery indicator.

   A. Indicator comes on if battery charge is low.

   B. Indicator also comes on if ionization voltage is too high.

FIELD MEASUREMENT OF PH, TEMPERATURE AND SPECIFIC CONDUCTIVITY IN WATER

1. Scope and Application

This method is applicable to in situ groundwater, monitoring well, residential, and municipal well samples, with measurement occurring at the sampling point.

2. Apparatus

The Corning M90 is a portable, microprocessor-based pH, specific conductivity, and temperature meter.

3. Reagents

A) pH reference buffer solutions:

1) pH = 4.00
2) pH = 7.00
3) pH = 10.00

B) Specific conductivity buffer solutions:
1) Conductivity standards A or B = 1413 μS or 12.88 mS

C) Distilled water

4. Calibration Procedures

A) Select sensor (i.e. pH, conductivity)
B) One-point calibration

1) Place the sensor in the calibrating medium:

Measurement	Solution	Reading
pH	pH = 7 buffer	7.00 pH (25°C)
Cond	Hold in free air	0.00 μS
TDS	Hold in free air	0.00 mg/L

2) Press CAL - cal 1 is displayed. After endpointing, the display automatically updates to the calibrated value shown, or the temperature compensated value.

3) If READ is pressed after cal 1 update, the meter assumes one point calibration only is required. Samples can now be measured.

C) Two-point calibration

1) Follow one point calibration. Place sensor in second calibrating medium:

Measurement	Solution	Reading
pH	pH 4 or pH 10 buffer	4.00 or 10.01 pH (at 25°C)
Cond	Cond std A or B	1413 uS or 12.88mS
TDS	Cond std A or B	706 mg/L or 6.44 g/L

2) Press CAL - cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown or the temperature compensated value.

5. Sample Handling and Preparation

Samples collected for pH, specific conductivity, and temperature should be obtained directly from the sampling point. Groundwater samples being tested during well purging can be obtained directly from the bailer.

6. Procedures

Select sensor for required measurement (i.e., pH sensor or conductivity sensor). Attach sensor to the M90 meter. Calibrate meter to the solution corresponding to type of sensor. Meter is now ready to make a measurement following these steps:

A) Prepare sensor

1) pH - Remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.

2) Specific conductivity/TDS - Immerse probe to halfway point in solution.

3) Temperature - pH and conductivity sensors automatically measure temperature.

B) Press MODE, READ, CAL, or M to turn on meter and start measurement. Place sensor into solution. Automatic endpoint detection freezes the display when plateau is reached; to manually endpoint press READ. Press READ again to start new measurement.

C) After use, close the fill hole and replace the wetting cap (pH).

7. Trouble-Shooting and Maintenance

A) Use distilled water when transferring from one solution to another.

B) Response time is a function of the sensor and the solution. If the solutions are at different temperatures (or ionic strength - pH only), allow more time for the sensor to respond.

C) Avoid handling the sensor tip.

D) Make sure no large air bubbles are trapped under the sensor when making measurements.

E) Do not use calibration standards after the expiration date.

F) Wetting caps should contain:
pH - pH 7 buffer.

G) For greatest accuracy callibrants and samples should be at the same temperature.

H) pH - Keep the electrode filled with the appropriate fill solution to prevent reading drift.

I) Conductivity - The sensor shield and probe should be kept clean. Make sure no air bubbles are in the cell chamber during measurement.

8. <u>Verification of Accuracy</u>

Following the last of the four replicate measurements, immerse the rinsed sensor in each of the reference solutions used to calibrate the meter/sensor prior to sample measurements. If the readings are not within 0.10 units of the reference values, recalibrate the meter/sensor and repeat the measurement of the sample just tested.

9. <u>Reporting</u>

A) pH - Report the average value of the replicate measurements to the nearest 0.1 units.

B) Temperature - Report the average value of the replicate measurements to the nearest 1°C.

C) Specific Conductivity - Report the average value of the replicate measurements to three significant digits.

APPENDIX C

STANDARD OPERATING PROCEDURES FOR COMPLETING THE CHAIN-OF-CUSTODY FORM

PROCEDURE FOR COMPLETING THE WESTON-ANALYTICS CHAIN-OF-CUSTODY FORM

The following information will be provided on the WESTON-Analytics chain-of-custody form.

Area 1

This section contains general project information.

Client: Techalloy RFI

Work Order No.: (A WESTON internal tracking number will be placed here.)

Project Contact/
Phone Number: Carlos Serna (708) 918-4002

Area 2

Sampling container packaging and preservation information is presented in this section.

Example: #/Type of Container: 1-glass; 2-P.E. (polyethylene)

Volume: 80 mL; 2 liters

Preservatives: HNO_3 (nitric acid), HCL (Hydrochloric Acid), NA (not applicable).

Area 3

Sample identification numbers are presented in this section.

Example: TC1-CP02-SBS-MS/MSD

Area 4

Quality control information is identified in this section. If the sample is assigned for MS/MSD analyses or spike/duplicate analyses, these columns are checked (✓ or X).

Area 5

The sample matrix is identified in this column.

Example: soil, water

Area 6

The date of sample collection will be presented in this section for each sample identified in Area 3.

Example: 4/16/93

Area 7

The time of sample collection for each sample identified in Area 3 will be specified in this column in military time.

Example: 1420 presenting 2:20 P.M.

Area 8

The required analyses/parameters are specified via a check mark (√ or X) in the appropriate box.

Area 9

Each required analyses/parameter being requested is identified in this section. Additional parameters can be added in the blank boxes.

Area 10

Any specific instructions or special information associated with the samples identified in Area 3 will be noted by the Field Sample Manager in this section.

Example:
- If the sample exhibited unusual characteristics (e.g.,odor, elevated PID readings).
- If the sample requires special handling at the laboratory.

Areas 11, 12, 13, 14

In these areas, the field person relinquishing the samples to the courier will acknowledge release of the samples. His/her signature will be placed in Area 11. Area 12 will remain blank. The relinquisher will state the date and time the samples left his/her custody in Areas 13 and 14, respectively.

Areas 15, 16, 17, 18

Upon receipt at the laboratory, the sample custodian logging in the samples will acknowledge receipt of the samples in these sections. Area 15 is left blank. The custodian's signature is placed in Area 16, and the time and date at which he/she acknowledges receipt of the samples will be stated in Areas 17 and 18, respectively.

Area 19

Following receipt of the sample shipment container(s), the laboratory sample custodian will check the samples received and complete this section based on his/her review.

WESTON Analytics Use Only

Custody Transfer Record/Lab Work Request

WESTON

Page ____ of ____

Client ____
Est. Final Proj. Sampling Date ____
Work Order # ____ 1
Project Contact/Phone # ____
AD Project Manager ____
QC ____ Del ____ TAT ____
Date Rec'd ____ Date Due ____
Account # ____

Refrigerator #
#/Type Container: Liquid / Solid
Volume: Liquid / Solid
Preservatives
ANALYSES REQUESTED →

2

ORGANIC: VOA, BNA, Pest/PCB, Herb

9

INORG: Metal, CN

WESTON Analytics Use Only

MATRIX CODES:
S - Soil
SE - Sediment
SO - Solid
SL - Sludge
W - Water
O - Oil
A - Air
DS - Drum Solids
DL - Drum Liquids
L - EP/TCLP Leachate
WI - Wipe
X - Other
F - Fish

Lab ID	Client ID/Description	Matrix QC Chosen (✓) MS	MSD	Matrix	Date Collected	Time Collected
	3	4		5	6	7

8

FIELD PERSONNEL: COMPLETE ONLY SHADED AREAS

Special Instructions:

10

DATE/REVISIONS:
1. ____
2. ____
3. ____
4. ____
5. ____
6. ____

WESTON Analytics Use Only

Samples were:
1) Shipped ___ or Hand Delivered ___
Airbill # ____
2) Ambient or Chilled
3) Received in Good Condition Y or N
4) Labels Indicate Properly Preserved Y or N
5) Received Within Holding Times Y or N

COC Tape was:
1) Present on Outer Package Y or N
2) Unbroken on Outer Package Y or N
3) Present on Sample Y or N
4) Unbroken on Sample Y or N
COC Record Present Upon Sample Rec't Y or N

19

Relinquished by	Received by	Date	Time	Relinquished by	Received by	Date	Time
11	12	13	14	15	16	17	18

Discrepancies Between Samples Labels and COC Record? Y or N
NOTES:

RFW 21-21-001/A-7/91 ____ L372 ____ L373 ____ L375 ____ L377 ____ L378 Ref# ____ Cooler# ____ 381 596

FIGURE

WESTON MANAGERS DESIGNERS/CONSULTANTS
Three Hawthorn Parkway
Vernon Hills, Illinois
60061

WESTON - ANALYTIC CHAIN-OF-CUSTODY FORM

TECHALLOY COMPANY, INC.
Union, Illinois

RWS-07/21/93-10:23-CAD93\SYMBOL\AH

APPENDIX D

STANDARD OPERATING PROCEDURES FOR ANALYTICAL METHODS
GULF COAST LABORATORIES, INC.

M. S. Iyer

INORGANIC ANALYSIS PROTOCOL
Analysis of Metals by Inductively Coupled Argon Plasma (ICAP) (Simultaneous Operation)

CONTROLLED DISTRIBUTION
COPY # : Uncontrolled
ISSUED TO : Techalloy CAPP
Full Signature Approvals Are Kept on File with WESTON®'s Analytics Division QA Standard Practice Records

REVISION NUMBER: 02

1.0 PURPOSE

To outline the guidelines for determining metal concentrations by Inductively Coupled Argon Plasma (ICAP) Emission Spectrometry - Simultaneous Operation.

2.0 REFERENCE

This SOP was written using USEPA SW-846 "Test Methods for Evaluating Solid Waste", Method 6010 as a reference.

3.0 METHOD SUMMARY

ICP is a technique for the analysis of solubilized or digested samples for metal concentrations using atomic emission spectrometry. All matrices including water, TCLP extracts, wastes, soils, sludges and sediments require digestion prior to analysis. The digestion procedures are listed in separate SOPs. The most common digestion procedures are SW-846 Methods 3010 and 3050. The instrument (Simultaneous ICP) is capable of analyzing simultaneously 29 different elements on a sample.

3.1 Parameter List

Element	Wavelength (nm) ICAP 1100	Reporting Limit (ug/L)
Ag	328.0	30
Al	308.2	200
As	193.6	100
B	249.6	50
Ba	493.4	50
Be	313.0	5
[1]Ca	315.8	100
Cd	228.8	5
Co	228.6	20
Cr	267.7	20
Cu	324.7	20
Fe	259.9	30
K	766.4	2000
Li	670.7	100
Mg	279.0	200
Mn	257.6	10
Mo	202.0	100
[1]Na	589.5	200
Ni	231.6	20
P	214.9	200
Pb	220.3	50
Sb	206.8	100
Sc	261.4	*
Se	196.0	100
[1]Si	288.1	100
Sn	189.9	100
Sr	421.5	100
Ti	334.9	10
[1]Tl	377.5	500
V	292.4	10
Y	371.0	*
Zn	213.8	10

* Sc is used as an internal standard on ICP 61.
* Y is used as an internal standard on ICP 1100.

[1]Alternate Wavelengths are used for ICAP 61:

Ca 317.9 Si 251.6
Na 588.9 Tl 190.8

The reporting limits that are listed above are at a level where the ICP can accurately quantitate at a concentration five times that listed.

Actual instrument detection limits (IDL) and linear ranges are also determined quarterly. The IDLs are determined by analyzing a solution at a concentration of five times the reporting limit (listed above) seven times on three non-consecutive days. The IDL is then calculated as 3 times the average standard deviation of these readings. The actual IDLs are always below the reporting limits.

4.0 INTERFERENCES

Spectral, Physical and Chemical Interferences are the three main interferences that are commonly present on the ICP.

4.1 Spectral Interferences

Mainly caused by continuous background wavelength, stray light from a high concentration element or overlap of a spectral line from another element. The ICP can correct for the first two types of interferences by using background correction adjacent to the wavelength. Spectral overlap can be corrected by monitoring the interfering wavelength and computer correcting the results for the false concentration. The values used to correct are known as Inter-element Correction Factors or IEC's.

4.2 Physical Interferences

Usually associated with the sample uptake and nebulization processes. These interferences can usually be eliminated by using a peristaltic pump which assures a constant sample uptake rate. If a sample is extremely viscous or contains a very high dissolved solids concentration, a dilution of the sample may be required to assure a constant and smooth nebulization rate.

4.3 Chemical Interferences

Normally not significant on the ICP. These interferences include ionization effects and molecular compound formation. Chemical interferences are highly dependent on the sample matrix type and the element.

Most interferences can be corrected by ensuring a constant sample uptake rate and by using the correcting abilities of the computer. If severe interferences are suspected, an alternate method such as flame AA or graphite furnace AA can be used or to verify the ICP results.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests.

6.0 INSTRUMENTATION AND EQUIPMENT

The instruments used are the ICAP 1100 and the ICP 61 which are made by Thermo Jarrell Ash. The instruments are simultaneous ICP's which currently have 29 analytical wavelengths. Additional wavelengths may be added as required.

The instruments are operated via a HAST 386 computer system and Thermo Jarrell Ash software. They also come equipped with a peristaltic pump for sample uptake and an autosampler.

7.0 PREVENTIVE MAINTENANCE

The required preventive maintenance is listed in the preventive maintenance notebook which is kept at the instrument. All maintenance is recorded in this notebook along with the date and the signature of the analyst preforming the maintenance.

7.1 Daily Maintenance

Includes changing the pump winding for consistent sample uptake and a visible check of the waste container to make sure that it doesn't overflow.

7.2 Weekly Maintenance

Includes checking the air filters on the back of the instrument for excessive dust buildup, checking the tip of the torch for excessive buildup of material, and dusting the instrument.

7.3 Monthly Maintenance

Includes cleaning and checking the water recirculator for proper fluid level, cleaning the spray chamber and checking and replacing the printer ribbon as necessary.

The instruments are under a full service contract with the manufacturer for all major repairs.

8.0 STANDARDS AND QC SOLUTIONS

8.1 Stock Standards and QC solutions

All stock standards and QC solutions are purchased from an outside supplier in aqueous form. Two types of standards are used: single element and custom mixed standards. The suppliers that are currently used are Inorganic Ventures, and SPEX. Single element standards are available for most elements at a 1000 mg/L concentration.

The shelf life of all purchased solutions is one year from receipt.

8.2 Calibration Standards

Prepared with Milli-Q water that has been acidified to the same concentrations as the samples. All calibration standards are spiked with 10 ppm of internal standard. The calibration standards are prepared daily as follows:

8.2.1 Calibration Blank

Add approximately 500 mL of Milli-Q DI water to a 1000 mL Class A volumetric flask. Repipette 10 mL of concentrated nitric acid and 50 mL of concentrated hydrochloric acid into the flask. Bring up to volume with Milli-Q DI water and mix thoroughly.

8.2.2 Calibration Standard

Add approximately 50 mL of Milli-Q deionized water to a 100 mL Class A volumetric flask. Re-pipette 1 mL of nitric acid and 5 mL of hydrochloric acid into the flask. Using Eppendorf pipettes, add 1.0 mL of XWGC-9, 1.0 mL XWGC-10A, 1.0 mL XWGC-11 and 1.0 mL PLAL2-3X. Bring up to volume with Milli-Q water and mix thoroughly.

* See Appendix A for element concentrations.

8.3 QC Solutions

Prepared with Milli-Q water that has been acidified to the same concentration as the samples. All QC solutions are spiked with 10 ppm of internal standards. The QC solutions are prepared as required (at least weekly) as follows:

8.3.1 ICV/CCV: Initial and Continuing Calibration Verification

Prepared by adding approximately 250 mL of Milli-Q water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of hydrochloric acid. Add 5.0 mL WGC-CAL-1C, 5.0 mL WGC-CAL-1D, and 25 mL of Al (1000 ug/mL). Bring up to volume with Milli-Q water and mix thoroughly.

* See Appendix B for element concentrations.

8.3.2 CRI

Prepared by adding approximately 250 mL of Milli-Q water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of concentrated hydrochloric acid to the flask. Add 5.0 mL WGC-CRI-1, and 5.0 mL of WGC-CRI-2. Bring up to volume with Milli-Q water and mix thoroughly.

* See Appendix C for element concentrations.

8.3.3 50% and 20% Standards

Prepared by making a serial dilution of the stock calibration standards. These solutions are used to verify the linearity of the calibration curve.

8.3.4 ICSA: Interferant Check Standard

Prepared by adding 250 mL of Milli-Q deionized water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of concentrated hydrochloric acid. Add 50 mL of CLPP-ICS-A. Bring up to volume with Milli-Q water and mix the solution thoroughly.

8.3.5 ICSAB: Interferant Check Standard

Prepared by adding 250 mL of Milli-Q deionized water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of concentrated hydrochloric acid. Add 50 mL of CLPP-ICS-A, 5.0 mL CLPP-ICS-B, 2.5 mL As (1000 ppm) and 2.5 mL Se (1000 ppm). Bring up to volume with Milli-Q water and mix thoroughly.

*See Appendix D for element concentrations for ICSA and ICSB Solutions.

9.0 PROCEDURE

9.1 Sample Preparation

The samples are digested according to the SOPs for the specific matrix type. The most commonly used digestion procedures are from EPA SW-846 Methods 3010 and 3050. The samples are checked for the proper preservation prior to digestion. The pH of the samples is checked using pH paper and the results (< or > 2) are recorded in the digestion notebook.

Prior to analysis all samples are spiked with 10 ppm of either Y or Sc (internal standard) depending on the instrument the samples will be analyzed on.

9.2 Instrument Set Up

Set up the instrument with the proper operating conditions as provided by Thermo Jarrell Ash. The instrument must be allowed to become thermally stable (usually requiring about an hour) prior to profiling and calibration.

Profile the instrument according to the manufacturer's recommended procedures

9.3 Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the linear range of the instrument.

The instrument is standardized using a calibration blank and calibration standard. The results are given in intensities.

9.4 Analytical Run

After the instrument is standardized, an analytical run is initiated. The first run of the day would proceed as follows:

STD	Reanalysis of calibration standard as a sample
ICV	Initial Calibration Verification
ICB	Initial Calibration Blank
CRI	Spiked Blank
ISA	Interferant Check Standard A
ISB	Interferant Check Standard B
20% STD	
50% STD	
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
PB	Preparation Blank
LC1	Laboratory Control Sample
LC2	Laboratory Control Sample
Sample 1	
Sample 1	Duplicate (D)

Sample 1	Spike (S)
Sample 1	Serial Dilution (L)
Sample 2	
.	
.	
Sample 10	
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank

* See Section 11.5 for acceptance criteria.

9.4.1 If the CCV and CCB results are acceptable, the run may continue without restandardization. If any of the post run QC is out of control, or close to being out of control, the instrument is restandardized before analyzing the next digestion. Any Samples with elements associated with an out of control CCV or CCB will be reanalyzed.

9.4.2 The CCV and CCB are run after every standardization, at a frequency of 10% during a run, and at the end of each analytical run.

9.4.3 The STD, 50% and 20% Standards are analyzed only at the beginning of each day. The CRI, ICSA and ICSB will be run at the beginning of the day and every 8 hours thereafter.

10.0 CALCULATIONS

The calculations for weight, volume, and dilution factors are performed by LIMS. The Sample ID is inputed for each sample. If a dilution is needed it is recorded on the run log and the dilution is put into LIMS via the data file.

Spreadsheets containing initial weight/volume and final volume along with % solids (if required) are updated in the LIMS system and this information is used to calculate results.

The sample results are stored in a data file on the HAST Computer. These files are then edited for any excess data or results, such as elements that were not required for a given sample. The edited file is then transferred directly to the LIMS system. This system helps to eliminate transcription errors, since data is not entered by hand.

10.1 Accuracy

10.1.1 ICV/CCV, LCS/LCSD % Recovery

$$\% R = \frac{\text{observed concentration}}{\text{actual concentration}} \times 100$$

10.1.2 Matrix Spike % Recovery

$$\% R = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

10.2 Precision

10.2.1 Matrix Dup. and LCS Dup. Relative Percent Difference (RPD)

$$RPD = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

11.0 QUALITY CONTROL

11.1 At least one Preparation Blank (PB) and at least two Laboratory Control Standards (LCS) will be included in each digestion batch of 20 samples. Regardless of the matrix being processed, the LCS and PB will be in an aqueous media. The Preparation Blanks are analyzed to determine if contaminants are being introduced into the sample via the Sample Preparation Procedures.

11.2 The Laboratory Control Samples are analyzed to determine both the precision and accuracy of the digestion process.

Precision will be measured by the reproducibility of both LCS results and will be calculated as Relative Percent Difference (RPD). Results must have a $\leq$ 20% RPD in order to be considered acceptable. If the LCS results are not within the control limits, all samples in the preparation set must be redigested and reanalyzed.

Statistical control limits are available and can be used per client request.

Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within +/- 20% of the known concentration. If either of the LCS results are outside the control limits, all samples in the preparation set must be redigested and reanalyzed.

* See Appendix E for element concentrations.

11.3 A duplicate sample will be prepared at a frequency of 5% (1 in 20 samples). RPD must be ±20%.

11.4 A spike sample will be prepared at a frequency of 5% (1 in 20 samples). Spike Recovery must be within 75 - 125%.

* See Appendix E for element concentrations.

11.5 A Serial Dilution (4X) will be prepared from the digestate at a frequency of 5% (1 in 20 samples). If the concentration is >10 times the IDL, results should agree within +/- 10% of the original results.

11.6 The acceptance limits for each solution are summarized below:

STD	±5% of the known concentration
ICV	±10% of the known concentration
ICB	+/- reporting limit
CRI	No limits have been established
50% and 20%	The linear regression curve for the high standard, 50% and 20% standards must have a correlation coefficient of ≥ 0.995.
ICSA	±20% of the known concentration
ICSB	±20% of the known concentration
CCV	±10% of the known concentration

CCB	+/- reporting limit
Matrix Dup.	If ≥ 5X reporting limit 20% RPD, if < 5X reporting limit ± reporting limit, if < reporting limit no control range
Matrix Spike	If sample concentration is ≤ 4X spike level 75-125%, if sample concentration is > 4X spike level no control range. If TCLP matrix spike is < 50%, Standard Addition must be performed.
Serial Dilution	If the analyte concentration is >10X the IDL, results should agree within +/- 10% of the original sample result.
LCS	80-120% of the known concentration
PB	± reporting limit

Currently, the acceptance criteria are at fixed control limits. Statistical limits are also produced by the QC department and are used when specified by the client or contract.

11.6 Metals Standards Traceability

Custom made and single element stock standard solution which are traceable to NIST or EPA are purchased. On receipt, each standard is recorded in a bound standards log book and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are documented in the log book.

12.0 CORRECTIVE ACTION

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the section manager.

12.1 Listed below are steps to be taken when an out of control situation occurs. The analyst must:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a corrective action report form;
- document on the corrective action report that an in control has been achieved; and
- receive approval (signature) of the unit leader, QA personnel, or the section manager prior to the release of any analytical data associated with the problem.

12.2 Suggested Actions to specific out of control situations:

12.2.1 Calibration Curve

- reanalyze the standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

12.2.2 Initial Calibration Verification (ICV)

- repeat ICV to verify proper preparation;
- prepare new ICV from original stock;
- recalibrate with a new standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

12.2.3 Initial Calibration Blank (ICB)

- prepare new ICB to verify proper preparation;
- verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability;
- determine the source of contamination by process of elimination, carryover from a previous analysis or reagent contamination and correct the problem;
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze the ICB and any associated samples.

12.2.4 Laboratory Control Standards (LCS)

If the LCS is low:
- reanalyze the LCS to verify that it is out of control;
- determine the source of error within the preparation procedure, repeat the sample set, write a CAR.

If the LCS is high:
- reanalyze the LCS to verify that it is out of control;
- determine the source of error within the preparation procedure, repeat the sample set;
- determine if the high value is due to contamination;
- check for contamination of reagents, LCS stock solution, or in the preparation area;
- correct for contamination, reanalyze;
- Write a CAR.

12.2.5 Laboratory Control Standard Duplicate (LCSD)

- Must meet all requirements and control limits as the LCS in addition to limits set for precision.

- Precision: if precision is out of control, initiate the same actions specified for the LCS.

12.2.6 Preparation Blank (PB)

· reanalyze the PB to verify that it is beyond the detection limit;
· determine the source of contamination;
· determine if a high value is due to contamination;
· check for contamination of reagents or in the preparation area;
· correct for contamination, reanalyze the set;
· in the extreme case where all samples in the set are at least ten times greater than the PB, reanalysis will not be required; however, a corrective action report and approval will be necessary.

12.2.7 Matrix Duplicate (DUP)

· a CAR will be written and approved by the Unit Leader. Sample will be ticked with a "*."

12.2.8 Matrix Spike (MS)

· A CAR will be written and approved by the Unit Leader. Sample will be ticked with a "*."

12.2.9 Serial Dilution (L)

· prepare a new serial dilution to verify proper preparation;
· a car will be written and approved by the unit leader or section manager.

12.2.10 Continuing Calibration Verification (CCV)

· repeat the CCV to verify proper preparation;
· prepare new a CCV from the original stock;
· check for instrument base-line drift or a change in one or more of the reagents;
· check reagents/solutions and prepare fresh if necessary;
· recalibrate with a new standard curve and repeat all samples since the previous in control CCV;
· never dispose of any samples until you are sure that all QC are within the control limits.

12.2.11 Continuing Calibration Blank (CCB)

- prepare a new CCB to verify proper preparation;
- verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc., to achieve stability;
- determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct problem;
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze the CCB and any associated samples;
- never dispose of any samples until you are sure that all QC are within the control limits.

If any of the ICV, ICB, Spike Blank, 50% standard, 20% standard, CCV or CCB results are out of control for any element, the instrument is restandardized and the samples associated with the out of control elements are reanalyzed.

If the PB or LCS are out of control for any element, the samples are redigested. An exception is if the sample concentrations are $\geq$ 10X the PB contamination. In this case, the results are reported as is.

If any of the Matrix Duplicate or Matrix Spike results are out of control, the client is notified of the poor results via a case narrative that is sent with the data report.

Corrective Action Report (CAR) forms are available for poor PB, LCS, matrix dup and matrix spike problems. These forms are completed by the analyst performing the analysis. The forms are then reviewed and signed by the unit leader. The signed forms are kept on file in the QC department.

Additional, detailed information on corrective action procedures is listed in Appendix B.

13. HEALTH AND SAFETY

Material Safety Data Sheets are available for all reagents.

Acids should be handled with care.

The standards contain potentially harmful elements. Care should be taken to avoid contact with the stock solutions. Wash hands well if contacted.

The ICP torch puts out harmful ultraviolet radiation. The torch should never be looked at directly without proper eye protection.

APPENDIX A

Standard Stock Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	Calibration Std. Conc. (mg/L)
Spex	XWGC-9	B	1000	10
		Ba	2000	20
		Be	500	5
		Ca	10000	100
		K	10000	100
		Li	1000	10
		Mg	10000	100
		Na	10000	100
		Se	1000	10
		Sr	1000	10
Spex	XWGC-10A	As	1000	10
		Pb	1000	10
		Sb	200	2
		Si	1000	10
		Sn	1000	10
		Tl	2000	20
Spex	XWGC-11	Ag	200	2
		Cd	500	5
		Co	1000	10
		Cr	1000	10
		Cu	1000	10
		Fe	5000	50
		Mn	1000	10
		Mo	1000	10
		Ni	1000	10
		Ti	1000	10
		V	1000	10
		Zn	1000	10
Spex	PLAL2-3x	Al	10000	100

APPENDIX B

Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICV/CCV Conc. (mg/L)
Inorganic Ventures	WGC-CAL-1C	Ca	5000	50
		Mg	5000	50
		K	5000	50
		Na	5000	50
		Fe	2500	25
		Ba	1000	10
		Tl	1000	10
		As	500	5
		Cr	500	5
		Co	500	5
		Cu	500	5
		Pb	500	5
		Li	500	5
		Mn	500	5
		Ni	500	5
		Se	500	5
		Sr	500	5
		V	500	5
		Zn	500	5
		Be	250	2.5
		Cd	250	2.5
		Ag	100	1
Inorganic Ventures	WGC-CAL-1D	B	500	5
		Mo	500	5
		Si	500	5
		Sn	500	5
		Ti	500	5
		Sb	100	1
Inorganic Ventures	Aluminum	Al	1000	50

APPENDIX C

Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	CRI Conc. (mg/L)
Inorganic Ventures	WGC-CRI-1	Ca	200	2
		Mg	200	2
		K	200	2
		Na	200	2
		Al	40	0.4
		Ba	40	0.4
		Tl	40	0.4
		As	20	0.2
		Fe	20	0.2
		Li	20	0.2
		Se	20	0.2
		Sr	20	0.2
		Co	10	0.1
		Pb	10	0.1
		V	10	0.1
		Ni	8	0.08
		Cu	5	0.05
		Zn	4	0.04
		Mn	3	0.03
		Cr	2	0.02
		Ag	2	0.02
		Be	1	0.01
		Cd	1	0.01
Inorganic Ventures	WGC-CRI-2	B	20	0.2
		Mo	20	0.2
		Si	20	0.2
		Sn	20	0.2
		Ti	20	0.2
		Sb	12	0.12

APPENDIX D

Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICSA Conc. (mg/L)
Inorganic Ventures	CLPP-ICS-A	Al	5000	500
		Ca	5000	500
		Mg	5000	500
		Fe	2000	200

				ICSB Conc. (mg/L)
Inorganic Ventures	CLPP-ICS-A	Al	5000	500
		Ca	5000	500
		Mg	5000	500
		Fe	2000	200
Inorganic Ventures	CLPP-ICS-B	Ag	100	1
		Cd	100	1
		Ni	100	1
		Pb	100	1
		Zn	100	1
		Ba	50	0.5
		Be	50	0.5
		Co	50	0.5
		Cr	50	0.5
		Cu	50	0.5
		Mn	50	0.5
		V	50	0.5
Inorganic Ventures	Arsenic	As	1000	5
Inorganic Ventures	Selenium	Se	1000	5

APPENDIX E
Known Digested QC Values
(mg/L)

Element	LCS/Spike	TCLP Spike
Al	2	-
Sb	0.5	-
As	2	5
Ba	2	100
Be	0.05	-
B	1	-
Cd	0.05	1
Ca	10	-
Cr	0.2	5
Co	0.5	-
Cu	0.25	-
Fe	1	-
Pb	0.5	5
Li	0.5	-
Mg	10	-
Mn	0.5	-
Mo	1	-
Ni	0.5	-
K	10	-
Se	2	1
Si	5	-
Ag	0.05	1
Na	10	-
Sr	1	-
Tl	2	-
Sn	1	-
Ti	1	-
V	0.5	-
Zn	0.5	-
P	10	-

<u>Control Limits</u>

LCS: 80 - 120%
Spike: 75 - 125%
TCLP Spike: >50%

RELEASED
2019-007454
July 27, 2020 - TJW

INORGANIC ANALYSIS PROTOCOL
Analysis of Metals by Inductively Coupled Argon Plasma (ICAP)
(Simultaneous Operation)

These Approval Signatures Are Kept on File
with WESTON®'s Analytics Division
QA Standard Practice Records

REVISION NUMBER: 02

	Printed Name:	Signature/Date:
Written By:	Jodi Wojcik ICP/Metals Preparation Unit Leader	Jodi L. Wojcik 7/13/93
Approved By:	Mani S. Iyer Metals Section Manager	Mani S. Iyer 7/13/93

Historical File: Revision 00: 08/28/90
Revision 01: 02/05/93
Revision 02: 07/13/93

Reasons for Change, Revision 02:

- Serial dilutions performed per each analytical batch
- Silicon spike concentration changed from 1 mg/L to 5 mg/L (Appendix E)

RELEASED
2019-007454
July 27, 2020 - TJW

INORGANIC ANALYSIS PROTOCOL
Total and Volatile Suspended Solids (Non-Filterable Residue)

CONTROLLED DISTRIBUTION
COPY # : Uncontrolled
ISSUED TO : Techalloy DAPP
Full Signature Approvals Are Kept on File with WESTON®'s Analytics Division QA Standard Practice Records

REVISION NUMBER: 01

1.0 PURPOSE

To determine the total non-filterable residue content in drinking, surface, and ground waters, domestic and industrial liquid wastes.

2.0 REFERENCE

This SOP was written using the following methods as references:

For Total Suspended Solids (TSS), EPA 600/4-79-020, Method 160.2 and Standard Methods, 17th Ed., Method 2540D.

For Volatile Suspended Solids (VSS), EPA 600/4-79-020, Method 160.4 and Standard Methods, 17th Ed., Method 2540E.

3.0 METHOD SUMMARY

3.1 Suspended solid matter in natural or man-made water bodies is of environmental importance because the presence of such material decreases the amount and depth of light penetration to the water body. The decrease in light penetration then decreases the potential for productivity of zooplankton and photoplankton of the water body, limiting the available food sources to higher vertebrates and invertebrates that are necessary to keep the ecosystem in balance.

3.2 Wastewater treatment plant operators use this information as a guide to pump performance within the plant and can often troubleshoot potential future problems. Final effluents are monitored to evaluate overall plant performance as well as to provide estimations of the suspended solids loading to the receiving stream.

3.3 Suspended solids material occurs naturally as silt, leaves, etc., and is caused by a variety of naturally occurring processes such as rain, decay, animal activities, etc. Litter and industrial effluents are sources of manmade suspended solids pollutants. Suspended solids materials are easily removed by filtration.

3.4 A sample may contain two types of solids, dissolved solids, and suspended solids. The suspended solids are those particles retained by a micro-fiber filter while the dissolved solids pass through the filter. The type of filter holder, the pore size, porosity, area and thickness of the filter and the physical nature, particle size and amount of material deposited on the filter are the principal factors affecting the trapping of suspended solids materials. The residue remaining on the filter paper is dried to a constant weight at 103-105°C.

If the TSS is less than the reporting limit, then VSS will not need to be determined.

4.0 INTERFERENCES

Exclude large floating particles or submerged agglomerates of non-homogeneous materials from the sample if they are not representative of the sample. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg of residue. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to excessive solids caught on the clogged filter.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Holding time, preservation techniques and sample container may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times, and the references which include container and preservation requirements for compliance with the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA).

Regulation	Holding Time	Reference
CWA NPDES	7 days	CFR 40 pt. 36.3
SDWA	7 days	EPA-570/9-82-002

All analyses are performed on unpreserved samples.

6.0 INSTRUMENT AND EQUIPMENT

- Analytical Balance - capable of weighing to 0.1 mg
- 103-105°C oven
- desiccator
- vacuum
- 100 mL graduated cylinder
- suction flask (side armed)
- crucible holder
- gooch crucibles, 40 mL
- Whatman 934-AH glass fiber filters
- 10 mL class A pipettes

7.0 PREVENTATIVE MAINTENANCE

7.1 Calibration check of balance, daily

7.2 Calibration check of 103-105°C oven, daily

7.3 Desiccant is inspected daily by analyst

8.0 STANDARDS AND REAGENTS

All standards and reagents are prepared with Type II Deionized Water **unless otherwise stated.**

8.1 Stock Solution I; 200 mg/L

Weigh out 200 mg diatamaceous earth (DE) to the nearest 1 mg and add it to 500 ml DI water in a 1.0 L volumetric flask. Dilute to volume with DI water and mix thoroughly.

- Life of QC Solution: one day
- Storage Requirements: none

8.2 Stock Solution II; 25 mg/L

Prepare as above using 25 mg DE rather than 200 mg.

- Life of QC Solution: one day
- Storage Requirements: none

9.0 PROCEDURE

9.1 Reporting Limit..................5 mg/L (based on a 100 mL minimum sample)

9.2 Sample Size

Working sample size varies, generally 100 ml is sufficient, but it is acceptable to increase or decrease the volume to obtain a range of at least 1.0 mg of residue, not to exceed 200 mg.

9.3 Crucible Preparation |

9.3.1 Assemble the filtering apparatus. Insert one filter disk into a crucible with the wrinkled side up and place the crucible in a vacuum apparatus. Wet the filter with a small volume of distilled water to seat it against the support. Turn on the vacuum and wash with three successive 20 mL volumes of DI water.

Continue suction until all traces of water are gone. Turn off the vacuum and place the crucible on the rack. When all crucibles are done, place the rack in a 103-105°C oven for a minimum of one hour.

After removing the rack from the oven, store it in a desiccator until cooled (a minimum of one hour). The moisture content will equilibrate during cooling.

9.3.2 To prepare a crucible for VSS, bring the muffle furnace up to 550 +/- 50°C. Place the prepared gooch(es) into the preheated muffle furnace for 15 minutes. Remove the gooch(es) and allow them to cool to room temperature before putting them into a desiccator. Store the gooch(es) in the desiccator for at least one hour prior to use.

9.3.3 Weigh the crucible and record the weight and crucible number.

9.4 Sample Analysis

9.4.1 Shake the sample vigorously and quantitatively transfer the sample to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

9.4.2 With suction still on, rinse the graduated cylinder with 10 mL of DI water and add that rinsate to the filter. Apply the vacuum until all the water is drained through. Rinse the filter with 2 additional 10 mL portions of DI water, allowing complete drainage between rinses. Apply suction for about 3 minutes after filtration is complete.

9.4.3 Carefully remove the crucible from the suction apparatus. Dry at least one hour at 103-105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg, or less than 4% of the previous weight, whichever is less.)

9.4.4 If VSS is to be determined, transfer the crucibles from 9.4.3 to a muffle furnace that has been pre-heated to 550 +/- 50°C and ignite for 15-20 minutes. Cool somewhat first then cool to the balance temperature in a desiccator. Weigh to the nearest 0.1 mg. Repeat the cycle of igniting, cooling and weighing until the weight loss is less than 4$ of the previous weight.

9.5 Analytical Sequence

Quality Controls	Frequency	Control Limit
Prep. Blk. (PB)	1 in 20 samples	< Reporting Limit
Lab. Control. Std. (LCS)	1 in 20 samples	+/- 20% Recovery
Lab. Control. Std. Dup. (LCSD)	1 in 20 samples	+/- 20% Recovery
Matrix Spike (MS)	1 in 20 samples	+/- 25% Recovery
Matrix Spike Dup. (MSD)	1 in 20 samples	+/- 20 RPD

▸All acronyms are defined in Section 12 of this procedure.
▸Drinking water samples must be analyzed in sets of 10 and have a matrix spike and duplicate performed on this matrix. The control limits for LCS's are +/- 10%; Matrix Spikes are +/- 15%; and Matrix Duplicates are $\leq$ 10 RPD.

9.7 Calibration Procedure

Laboratory balances are calibrated and serviced annually by a factory representative. In addition, an analyst checks the balance daily with two masses: one in the gram range and one in the milligram range. A record of calibrations and daily checks will be kept in the balance log.

Class S weights are used by the analysts for daily balance checks.

Oven and refrigerator thermometers will be calibrated annually against a National Bureau of Standards (NBS) certified thermometer in the range of interest. Annual calibrations will be recorded in a calibration notebook. Daily readings will be recorded with the respective oven or refrigerator before and after the drying period.

9.7.1 Standards Preparation

9.7.1.1 Preparation Blank (PB)

300 mls DI water

9.7.1.2 Laboratory Control Standard (LCS) and LCS Duplicate; 200 mg/L

300 mLs of Stock Solution I (Rgt. 8.1).

9.7.1.3 Matrix Spike; 25 mg/L

Pour 100 mLs of Stock Solution II (Rgt. 8.2) through the same filter as the sample to be spiked. If possible (see constraints from Section 4.0), spike 100 mL of sample for a spike concentration of 25 mg/L. If other than 100 mL of sample volume was spike, calculate the spike concentration as follows:

$$\text{mg/L spike concentration} = \frac{25 \text{ mg/L} \times 100 \text{ mL}}{\text{mL sample}}$$

NOTE: There is no matrix spike or LCS for VSS. Only a preparation blank and a matrix duplicate can be analyzed.

9.7.1.4 Matrix Duplicate

Duplicate of one sample.

10.0 CALCULATIONS

10.1 Sample Calculation

10.1.1 Total Suspended Solids

$$\text{mg/L} = \frac{(A - B) \times 1{,}000{,}000}{C}$$

Where:
A = final weight (g) of crucible plus sample
B = initial weight (g) of crucible
C = sample volume (mL)

10.1.2 Volatile Suspended Solids

$$\text{mg/L} = \frac{(A - D) \times 1{,}000{,}000}{C}$$

Where:
A = weight of crucible and residue before igniting (g)
D = weight of crucible and residue after igniting (g)
C = sample volume (mL)

10.2 Accuracy

10.2.1 LCS/LCSD % Recoveries

$$\%\,R = \frac{\text{observed concentration}}{\text{actual concentration}} \times 100$$

10.2.2 Matrix Spike % Recovery

$$\%\,R = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

10.3 Precision

10.3.1 Matrix Dup. and LCS Dup. Relative Percent Difference (RPD)

$$\text{RPD} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

10.4 Reporting Results

Without rounding, enter the raw data on the appropriate Lotus spreadsheet. Carefully print, review and approve the spreadsheet. Have the data book and spreadsheet approved and signed by the designated reviewer before creating a print file and transferring the data to LIMS.

11.0 QUALITY CONTROL

11.1 One method blank and two Laboratory Control Standards (LCS) will be included in each laboratory lot of 20 samples. Regardless of the matrix being processed, the LCS and method blanks will be in an aqueous media.

11.2 The method blank will be examined to determine if contamination is being introduced in the laboratory.

11.3 The LCS's will be examined to determine both precision and accuracy.

11.4 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within the laboratory's acceptance limits in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

11.5 Precision will be measured by the reproducibility of both LCS's and will be calculated as relative percent difference (%RPD). Results must agree within in-house control limits or statistical control limits in order to be considered acceptable.

11.6 One matrix spike and matrix duplicate is performed per matrix per 20 sample analytical set. Results must agree within the in-house precision/accuracy limits or statistical control limits in order to be considered acceptable.

12.0 CORRECTIVE ACTIONS

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the lab manager.

12.1 Listed below are steps to be taken when an out of control situation occurs. The analyst must:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a corrective action report form;
- document on the corrective action report that an in control has been achieved; and
- receive approval (signature) of the Section Manager, Unit Leader, QA personnel, or the Laboratory Manager prior to the release of any analytical data associated with the problem.

12.2 Suggested Actions to specific out of control situations:

12.2.1 Laboratory Control Standards (LCS)

If either LCS1 or LCS2 exceeds acceptance limits:
- reanalyze LCS to verify that an out of control situation exists;
- determine the source of error within the preparation procedure, correct the problem and repeat the sample set. (Sources of contamination could be either the reagents, the LCS stock solution, or the preparation area.)

Precision: LCS1 and LCS2 must meet the control limits of $\leq$ 20% RPD. If this criteria is not met, and both LCS's meet the % Recovery control limits, then see your Section Manager or Unit Leader for proper corrective action.

12.2.2 Preparation Blank (PB)

- reanalyze PB to verify contamination at a level > Reporting Limit;
- determine the source of contamination and correct the problem;
- all samples whose concentration is <10 times the PB level must be reprocessed and reanalyzed; any sample which is >10 times the PB level need not be reanalyzed. However, a corrective action report must be filled out and approval obtained.

12.2.3 Matrix Duplicate (DUP)

· the sample must be reprocessed and reanalyzed unless the sample concentration is <5 times the Reporting Limit, then the ± Reporting Limit rule applies;
· if the reanalysis is within the control limits, the second value is reported;
· if the reanalysis is still outside of the control limits, a CAR must be written and then approved by your Section Manager or Unit Leader.

12.2.4 Matrix Spike (MS)

· the sample must then be reprocessed and reanalyzed unless the sample concentration exceeds the spike concentration by a factor of 4 times;
· the original spike results must be entered onto the spreadsheet with the "S" code even though the control limits were exceeded;
· the reanalysis result must be entered onto the spreadsheet using the "T" code regardless of whether it is within the control limits. There is no need to write a corrective action if both the "S" and "T" codes were entered into LIMS.

13.0 HEALTH AND SAFETY

As always, general laboratory safety practices should always be followed. Waste samples should be handled with care due to the uncertainty of the properties and contents involved. Refer to the specific MSDS for the hazardous properties of any chemical or reagent involved in this procedure.

INORGANIC ANALYSIS PROTOCOL
Total and Volatile Suspended Solids
(Non-Filterable Residue)

These Approval Signatures Are Kept on File with WESTON®'s Analytics Division QA Standard Practice Records

REVISION NUMBER: 01

	Printed Name:	Signature/Date:
Updated By:	Donna L. Storm Wet Chemistry Analyst	Donna L. Storm 3/22/93
Contributor:	Diane L. Harper Wet Chemistry Section Manager	Diane L Harper 3-22-93
Approved By:	Diane L. Harper Wet Chemistry Section Manager	Diane L. Harper 3-22-93

Historical File: Revision 00: 07/20/90
Revision 01: 03/19/93

Reasons for Change, Revision 01:

- Addition of Method 160.4 to the text.
- Reformatted Section 9.0 for clarity.
- Added calculations for Volatile Suspended Solids.

APPENDIX E

SPECIFICATIONS AND GUIDANCE FOR OBTAINING CONTAMINANT-FREE SAMPLE CONTAINERS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MAY 2 1990

MEMORANDUM

SUBJECT: Revision of "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers"

FROM: Joan F. Fisk, Chief, Joan Fisk
Analytical Methods Implementation Section,
Analytical Operations Branch,
Hazardous Site Evaluation Division (OS-230)

TO: Addressees

In September, 1989 you received OSWER Directive #9240.0-05 from Henry Longest II with the memorandum titled "Decentralization of the Superfund Bottle Repository Functions". The purpose of this transmittal is to provide you with a revised version of the "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" that addresses problems brought up once the original document was put into use. This revised version has been through extensive review provided by the Regions through the Contract Laboratory Technical Project Officers who circulated the draft for comments.

The Analytical Operations Branch plans to transmit this document formally with an amended directive number, but since we have had so many urgent requests for it, we decided that this early distribution to you would be of great assistance in your procuring of bottles. We would appreciate any comments that you have as soon as possible, so that if we have overlooked any deficiencies we can remedy them prior to the transmittal as a directive.

Addressees:

Contract Laboratory Program Technical Project Officers
Regional Sample Control Centers
Superfund Branch Chiefs

cc:

Director, Waste Management Division
Regions I, IV, V, VII, VIII
Director, Emergency and Remedial Response Division
Region II
Director, Hazardous Waste Management Division
Regions III, VI
Director, Toxic and Waste Management Division
Region IX
Director, Hazardous Waste Division
Region X
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SPECIFICATIONS

AND

GUIDANCE

FOR OBTAINING

CONTAMINANT-FREE SAMPLE CONTAINERS

APRIL, 1990

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SECTION I

INTRODUCTION

In August 1989, the Environmental Protection Agency's (EPA) Office of Emergency and Remedial Response (OERR) decentralized Superfund's Sample Container Repository program (OSWER Directive #9240.0-05). In conjunction with the decentralization of Superfund's bottle program, OERR issued initial "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" (August 1989) to assist the Regions in obtaining appropriate sample containers from commercially available suppliers.

This document revises the initial specifications and provides a single source of standardized specifications and guidance on appropriate cleaning procedures for preparing contaminant-free sample containers that meet all Contract Laboratory Program (CLP) detection/quantitation limits, including those for newly established low concentration analyses. Although the specifications and guidance procedures contained in this document are based on CLP low concentration requirements, they also are suitable for use in other analytical programs. Specific needs of EPA Regions will dictate which cleaning procedures are used by the designated bottle preparer.

Major revisions in this document include:

- Allowing the use of polypropylene closures as an alternative to phenolic closures;
- Specifying the use of CLP Inorganic Low Concentration Contract Required Detection Limits (CRDL);
- Specifying the use of CLP Organic Low Concentration Contract Required Quantitation Limits (CRQL);
- Including procedures for the cleaning of containers for fluoride and nitrate/nitrite analyses;
- Including procedures for the quality control analysis of fluoride and nitrate/nitrite; and
- Specifying the use of CLP Inorganic and Organic Low Concentration analytical methods for quality control analyses.

OERR and the EPA Regions decided to use the most stringent CLP requirements available to set the specifications for obtaining contaminant-free sample containers. As a result, the CLP Inorganic and Organic Low Concentration Statement of Work (SOW) requirements were selected as the basis for these specifications. Major factors in this decision included the desire to have a single set of bottle cleaning specifications that met or exceeded all analytical requirements and the related need to avoid potential misuse of cleaned bottles (e.g., using a container cleaned by a multi-concentration procedure for a low concentration sample). OERR will reevaluate this decision if the low concentration requirements are deemed to be too stringent.

Specifications and guidance for preparing contaminant-free sample containers are provided in the sections that follow and are intended to describe one approach for obtaining cleaned, contaminant-free sample containers for use by groups performing sample collection activities under Superfund and other hazardous waste programs. Although other cleaning procedures may be used, sample containers must meet the criteria specified in Section II. In certain instances, the user of the sample containers may require exact adherence to the cleaning procedures and/or quality control analysis described in this document. In other instances, the user may require additional or different cleaning procedures and/or quality control analysis of the sample containers. The specific needs of the bottle user will determine the requirements for the cleaning and quality control analysis of the sample containers.

Most environmental sampling and analytical applications offer numerous opportunities for sample contamination. For this reason, contamination is a common source of error in environmental measurements. The sample container itself represents one such source of sample contamination. Hence, it is vital that sample containers used within the Superfund program meet strict specifications established to minimize contamination which could affect subsequent analytical determinations. Superfund sampling and analysis activities require all component materials (caps, liners, septa, packaging materials, etc.) provided by the bottle preparer to meet or exceed the criteria limits of the bottle specifications listed within Section II.

Section III provides guidance on cleaning procedures for preparing contaminant-free sample containers that meet the specifications contained in Section II. The procedures provided in this section are intended to provide sample containers that meet all current CLP Low Concentration Inorganic and Organic detection/quantitation levels.

In selecting cleaning procedures for sample containers, it is important to consider all of the parameters of interest. Although a given cleaning procedure may be effective for one parameter or type of analysis, it may be ineffective for another. When multiple determinations are performed on a single sample or on a subsample from a single container, a cleaning procedure may actually be a source of contamination for some analytes while minimizing contamination in others. It should be the responsibility of the bottle supplier to verify that the cleaning procedures actually used satisfy the quality control requirements set forth in Section IV.

Two aspects of quality assurance (i.e., quality control and quality assessment) must be applied to sample containers as well as to the analytical measurements. Quality control includes the application of good laboratory practices and standard operating procedures especially designed for the cleaning of sample containers. The cleaning operation should be based on protocols especially designed for specific contaminant problems. Strict adherence to these cleaning protocols is imperative.

Quality assessment of the cleaning process depends largely on monitoring for adherence to the respective protocols. Because of their critical role in the quality assessment of the cleaning operation, protocols must be carefully designed and followed.

Guidance is provided in Section IV on design and implementation of quality assurance and quality control protocols.

SECTION II

SAMPLE CONTAINER AND COMPONENT MATERIAL SPECIFICATIONS

This Section identifies sample containers commonly used in the Superfund program and provides specifications for contaminant-free sample containers for each bottle type.

A. CONTAINER MATERIAL

A variety of factors affect the choice of containers and cap material. These include resistance to breakage, size, weight, interferences with analytes of interest, cost, and availability.

Container types A through L (Figure 1, pages 6-7) are designated as the type of sample containers that have been used successfully in the past. Kimax or Pyrex brand borosilicate glass is inert to most materials and is recommended where glass containers are used (i.e., pesticides and other organics). Conventional polyethylene is recommended when plastic is acceptable because of its lower cost and lower adsorption of metal ions. The specific sampling situation will determine the use of plastic or glass.

While the sample containers shown in Figure 1 are utilized primarily for Superfund sampling activities, they also may be used for sampling activities under other programs, such as the Resource Conservation and Recovery Act (RCRA).

B. MAXIMUM CONTAMINANT LEVEL SPECIFICATIONS FOR SAMPLE CONTAINERS

The CLP, through a series of technical caucuses, has established inorganic Contract Required Detection Limits (CRDL) and organic Contract Required Quantitation Limits (CRQL) which represent the minimum detectable quantities needed to support the hazardous substance identification and monitoring requirements necessary for remedial and other actions at hazardous waste sites.

The philosophy used for determining the maximum permissible amount of contamination in a sample container was to consider the number of aliquots of sample that are available in the container and assume that the contamination present would be uniformly distributed in all of the aliquots. This assumption, and the assumption that there should be no more than one-half the CRDL or CRQL contributed by the container, resulted in the establishment of contamination limits by container type.

For inorganic sample containers, the CRDLs listed in Table 1, page 8, are the specifications for maximum trace metal contamination. Concentration at or above these limits on any parameter should preclude these containers from use in collecting inorganic samples.

The CRQL specifications for organic sample containers are listed in Table 2, pages 9-13. When the CRQL in Table 2 is multiplied by the appropriate factor listed below, the resulting value then represents the maximum concentration allowed for particular sample containers based on organic CLP sample sizes for routine analyses.

Container type	Multiple of CRQL
A	1.0
B	0.5
D	10.0
E	8.0
F	4.0
G	2.0
H	0.5
J	0.5
K	2.0

C. GROSS CONTAMINATION

Gross contamination is defined as greater than two hundred times the acceptable concentration values in Tables 1 or 2, unless the cleaning procedure is successful in reducing the amount of contamination to within specifications. If this is not achieved, the grossly contaminated materials should be discarded and replaced to prevent cross contamination with other batches of containers.

The bottle preparer should inspect all materials to ensure conformance with the required specifications.

FIGURE 1

SAMPLE CONTAINER SPECIFICATIONS

Container Type	Specifications
A	Container: 80-oz amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm teflon liner. Total Weight: 2.45 lbs.
B	Container: 40-mL glass vial, 24-mm neck finish. Closure: white polypropylene or black phenolic, open-top, screw cap, 15-cm opening, 24-400 size. Septum: 24-mm disc of 0.005-in teflon bonded to 0.120-in silicon for total thickness of 0.125-in. Total Weight: 0.72 oz.
C	Container: 1-L high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: white polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.89 oz.
D	Container: 120-mL wide mouth, glass vial, 48-mm neck finish. Closure: white polypropylene cap, 48-400 size; 0.015-mm teflon liner. Total Weight: 4.41 oz.
E	Container: 16-oz tall, wide mouth, straight-sided, flint glass jar, 63-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 63-400 size; 0.015-mm teflon liner. Total Weight: 9.95 oz.
F	Container: 8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 58-400 size; 0.030-mm teflon liner. Total Weight: 7.55 oz.

FIGURE 1

SAMPLE CONTAINER SPECIFICATIONS (Continued)

Container Type	Specifications
G	Container: 4-oz tall, wide mouth, straight-sided, flint glass jar, 48-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm teflon liner. Total Weight: 4.70 oz.
H	Container: 1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm teflon liner. Total Weight: 1.11 lbs.
J	Container: 32-oz tall, wide mouth, straight-sided, flint glass jar, 89-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 89-400 size; 0.015-mm teflon liner. Total Weight: 1.06 lbs.
K	Container: 4-L amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: white polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm teflon liner. Total Weight: 2.88 lbs.
L	Container: 500-mL high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: white polypropylene cap, white ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.20 oz.

TABLE 1

INORGANIC ANALYTE SPECIFICATIONS

	Analyte	Contract Required Detection Limits[1] (µg/L)
1.	Aluminum	100
2.	Antimony	5
3.	Arsenic	2
4.	Barium	20
5.	Beryllium	1
6.	Cadmium	1
7.	Calcium	500
8.	Chromium	10
9.	Cobalt	10
10.	Copper	10
11.	Iron	500
12.	Lead	2
13.	Magnesium	500
14.	Manganese	10
15.	Mercury	0.2
16.	Nickel	20
17.	Potassium	750
18.	Selenium	3
19.	Silver	10
20.	Sodium	500
21.	Thallium	10
22.	Vanadium	10
23.	Zinc	20
24.	Cyanide	10
25.	Fluoride	200
26.	Nitrate/Nitrite	100

1 CRDLs are based on the CLP Inorganic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND SPECIFICATIONS

	Volatiles	CAS Number	Contract Required Quantitation Limits[1] ($\mu g/L$)
1.	Chloromethane	74-87-3	1
2.	Bromomethane	74-83-9	1
3.	Vinyl Chloride	75-01-4	1
4.	Chloroethane	75-00-3	1
5.	Methylene Chloride	75-09-2	2
6.	Acetone	67-64-1	5
7.	Carbon Disulfide	75-15-0	1
8.	1,1-Dichloroethene	75-35-4	1
9.	1,1-Dichloroethane	75-34-3	1
10.	cis-1,2-Dichloroethene	156-59-4	1
11.	trans-1,2-Dichloroethene	156-60-5	1
12.	Chloroform	67-66-3	1
13.	1,2-Dichloroethane	107-06-2	1
14.	2-Butanone	78-93-3	5
15.	Bromochloromethane	74-97-5	1
16.	1,1,1-Trichloroethane	71-55-6	1
17.	Carbon Tetrachloride	56-23-5	1
18.	Bromodichloromethane	75-27-4	1
19.	1,2-Dichloropropane	78-87-5	1
20.	cis-1,3-Dichloropropene	10061-01-5	1
21.	Trichloroethene	79-01-6	1
22.	Dibromochloromethane	124-48-1	1
23.	1,1,2-Trichloroethane	79-00-5	1
24.	Benzene	71-43-2	1
25.	trans-1,3-Dichloropropene	10061-02-6	1
26.	Bromoform	75-25-2	1
27.	4-Methyl-2-pentanone	108-10-1	5
28.	2-Hexanone	591-78-6	5
29.	Tetrachloroethene	127-18-4	1
30.	1,1,2,2-Tetrachloroethane	79-34-5	1

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Volatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
31.	1,2-Dibromoethane	106-93-4	1
32.	Toluene	108-88-3	1
33.	Chlorobenzene	108-90-7	1
34.	Ethylbenzene	100-41-4	1
35.	Styrene	100-42-5	1
36.	Xylenes (total)	1330-20-7	1
37.	1,3-Dichlorobenzene	541-73-1	1
38.	1,4-Dichlorobenzene	106-46-7	1
39.	1,2-Dichlorobenzene	95-50-1	1
40.	1,2-Dibromo-3-chloropropane	96-12-8	1

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND SPECIFICATIONS (Continued)

	Semivolatiles	CAS Number	Contract Required Quantitation Limits[1] ($\mu g/L$)
1.	Phenol	108-95-2	5
2.	bis-(2-Chlorethyl)ether	111-44-4	5
3.	2-Chlorophenol	95-57-8	5
4.	2-Methylphenol	95-48-7	5
5.	2,2'-oxybis-(1-Chloropropane)	108-60-1	5
6.	4-Methylphenol	106-44-5	5
7.	N-Nitroso-di-n-dipropylamine	621-64-7	5
8.	Hexachloroethane	67-72-1	5
9.	Nitrobenzene	98-95-3	5
10.	Isophorone	78-59-1	5
11.	2-Nitrophenol	88-75-5	5
12.	2,4-Dimethylphenol	105-67-9	5
13.	bis-(2-Chloroethoxy)methane	111-91-1	5
14.	2,4-Dichlorophenol	120-83-2	5
15.	1,2,4-Trichlorobenzene	120-82-1	5
16.	Naphthalene	91-20-3	5
17.	4-Chloroaniline	106-47-8	5
18	Hexachlorobutadiene	87-68-3	5
19.	4-Chloro-3-methylphenol	59-50-7	5
20.	2-Methylnaphthalene	91-57-6	5
21.	Hexachlorocyclopentadiene	77-47-4	5
22.	2,4,6-Trichlorophenol	88-06-2	5
23.	2,4,5-Trichlorophenol	95-95-4	20
24.	2-Chloronaphthalene	91-58-7	5
25.	2-Nitroaniline	88-74-4	20
26.	Dimethylphthalate	131-11-3	5
27.	Acenaphthylene	208-96-8	5
28.	2,6-Dinitrotoluene	606-20-2	5
29.	3-Nitroaniline	99-09-2	20
30.	Acenaphthene	83-32-9	5

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND SPECIFICATIONS (Continued)

	Semivolatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
31.	2,4-Dinitrophenol	51-28-5	20
32.	4-Nitrophenol	100-02-7	20
33.	Dibenzofuran	132-64-9	5
34.	2,4-Dinitrotoluene	121-14-2	5
35.	Diethylphthalate	84-66-2	5
36.	4-Chlorophenyl-phenylether	7005-72-3	5
37.	Fluorene	86-73-7	5
38.	4-Nitroaniline	100-01-6	20
39.	4,6-Dinitro-2-methylphenol	534-52-1	20
40.	N-Nitrosodiphenylamine	86-30-6	5
41.	4-Bromophenyl-phenylether	101-55-3	5
42.	Hexachlorobenzene	118-74-1	5
43.	Pentachlorophenol	87-86-5	20
44.	Phenanthrene	85-01-8	5
45.	Anthracene	120-12-7	5
46.	Di-n-butylphthalate	84-74-2	5
47.	Fluoranthene	206-44-0	5
48	Pyrene	129-00-0	5
49.	Butylbenzylphthalate	85-68-7	5
50.	3,3'-Dichlorobenzidine	91-94-1	5
51.	Benz[a]anthracene	56-55-3	5
52.	Chyrsene	218-01-9	5
53.	bis-(2-Ethylhexyl)phthalate	117-81-7	5
54.	Di-n-octylphthalate	117-84-0	5
55.	Benzo[b]fluoranthene	205-99-2	5
56.	Benzo[k]fluoranthene	207-08-9	5
57.	Benzo[a]pyrene	50-32-8	5
58.	Indeno(1,2,3-cd)pyrene	193-39-5	5
59.	Dibenz[a,h]anthracene	53-70-3	5
60.	Benzo[g,h,i]perylene	191-24-2	5

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Pesticides/PCBs	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
1.	alpha-BHC	319-84-6	0.01
2.	beta-BHC	319-85-7	0.01
3.	delta-BHC	319-86-8	0.01
4.	gamma-BHC (Lindane)	58-89-9	0.01
5.	Heptachlor	76-44-8	0.01
6.	Aldrin	309-00-2	0.01
7.	Heptachlor epoxide	1024-57-3	0.01
8.	Endosulfan I	959-98-8	0.01
9.	Dieldrin	60-57-1	0.02
10.	4,4'-DDE	72-55-9	0.02
11.	Endrin	72-20-8	0.02
12.	Endosulfan II	33213-65-9	0.02
13.	4,4'-DDD	72-54-8	0.02
14.	Endosulfan sulfate	1031-07-8	0.02
15.	4,4'-DDT	50-29-3	0.02
16.	Methoxychlor	72-43-5	0.10
17.	Endrin ketone	53494-70-5	0.02
18	Endrin aldehyde	7421-36-3	0.02
19.	alpha-Chlordane	5103-71-9	0.01
20.	gamma-Chlordane	5103-74-2	0.01
21.	Toxaphene	8001-35-2	1.0
22.	Aroclor-1016	12674-11-2	0.20
23.	Aroclor-1221	11104-28-2	0.20
24.	Aroclor-1232	11141-16-5	0.40
25.	Aroclor-1242	53469-21-9	0.20
26.	Aroclor-1248	12672-29-6	0.20
27.	Aroclor-1254	11097-69-1	0.20
28.	Aroclor-1260	11096-82-5	0.20

1 CRQLs are based on the CLP Organic Low Concentration SOW (1990)